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**A DUAL VESSEL SYSTEM OF PHOSPHATING
FERROUS ALLOYS UNDER STEAM PRESSURE**

by
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and
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AUGUST 1979



ENGINEERING DIRECTORATE

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20. ABSTRACT (continued)

the bath can be used continuously on a production-type basis. Four types of manganese enriched baths were used in testing the dual vessel system. Salt spray corrosion testing was divided into two groups: unheated panels and panels heated to 450°F for one hour. Results from the manganese tartrate enriched bath best illustrated the success of pressure phosphating using consecutive production-type runs in the dual vessel system. For this manganese tartrate bath, 90% of the heated and 81% of the unheated sets of panels passed a minimum 100-hour salt spray resistance test. Continuous control of free (to less than 3.5 points) and total acid (to above 30 points) values and proper maintenance of the bath with chemical additions after each processing period are needed for reproducible superior heat and corrosion resistant phosphate coatings. A manufacturing level phosphating capacity and the design of a sludge recycling system is recommended for utilization of this process in the application of heavy manganese phosphate coatings to ferrous metal items. (U) (Crain, Henry and Wagner, Linden)

FOREWORD

The project was carried out under the title "Application of Heat and Corrosion Resistant Phosphate Coatings." This work was authorized as part of the Manufacturing Methods and Technology Program of the U.S. Army Materiel Development and Readiness Command and was administered by the U.S. Army Industrial Base Engineering Activity.

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INTRODUCTION

Manganese phosphate coatings are conventionally applied to ferrous articles by immersion in a phosphating bath operated at 200°F - 210°F. These coatings are required by Specification MIL-P-16232 to withstand a 1-1/2 hour salt spray test (ASTM B117).¹ The coatings are applied to military small arms weapon components when low reflectivity and corrosion resistance are desired. They also serve as a base for the supplementary application of lubricant for increased corrosion protection. At low temperatures, phosphate coatings retain their protective properties; however, at temperatures above 230°F, they deteriorate and show a large reduction in corrosion resistance.

Recently, a new and improved manganese phosphate coating has been developed in this Laboratory. The new method of phosphating produces superior corrosion resistant coatings capable of withstanding temperatures up to 450°F. The coating can also withstand the 5% salt spray test for more than five-hundred hours. This represents at least a hundred-fold increase in corrosion protection over that of conventional manganese phosphate coatings. The new method of phosphating requires bath temperatures above 215°F using steam pressures in excess of 1 psig and additions to the bath of either manganese citrate, manganese tartrate, or manganese gluconate. A previous report² on phosphating using steam pressure, describes a batch processing procedure. The report on the dual vessel system describes the continuous production of superior manganese phosphate coatings on a pilot line.

PROCEDURE

The apparatus for producing superior phosphate coatings consists of dual vessels shown in Figure 1. This system has the following capabilities:

- a. The phosphating bath can be transferred from one vessel to another under differential steam pressure.

¹Annual Book of ASTM Standards, American Society for Testing and Materials, Vol. 7, 66, 1970.

²Wagner, Linden H., "The Application of Heat and Corrosion Resistant Phosphate Coatings Under Steam Pressure," Rodman Laboratory Technical Report R-TR-74-012, March 1974.

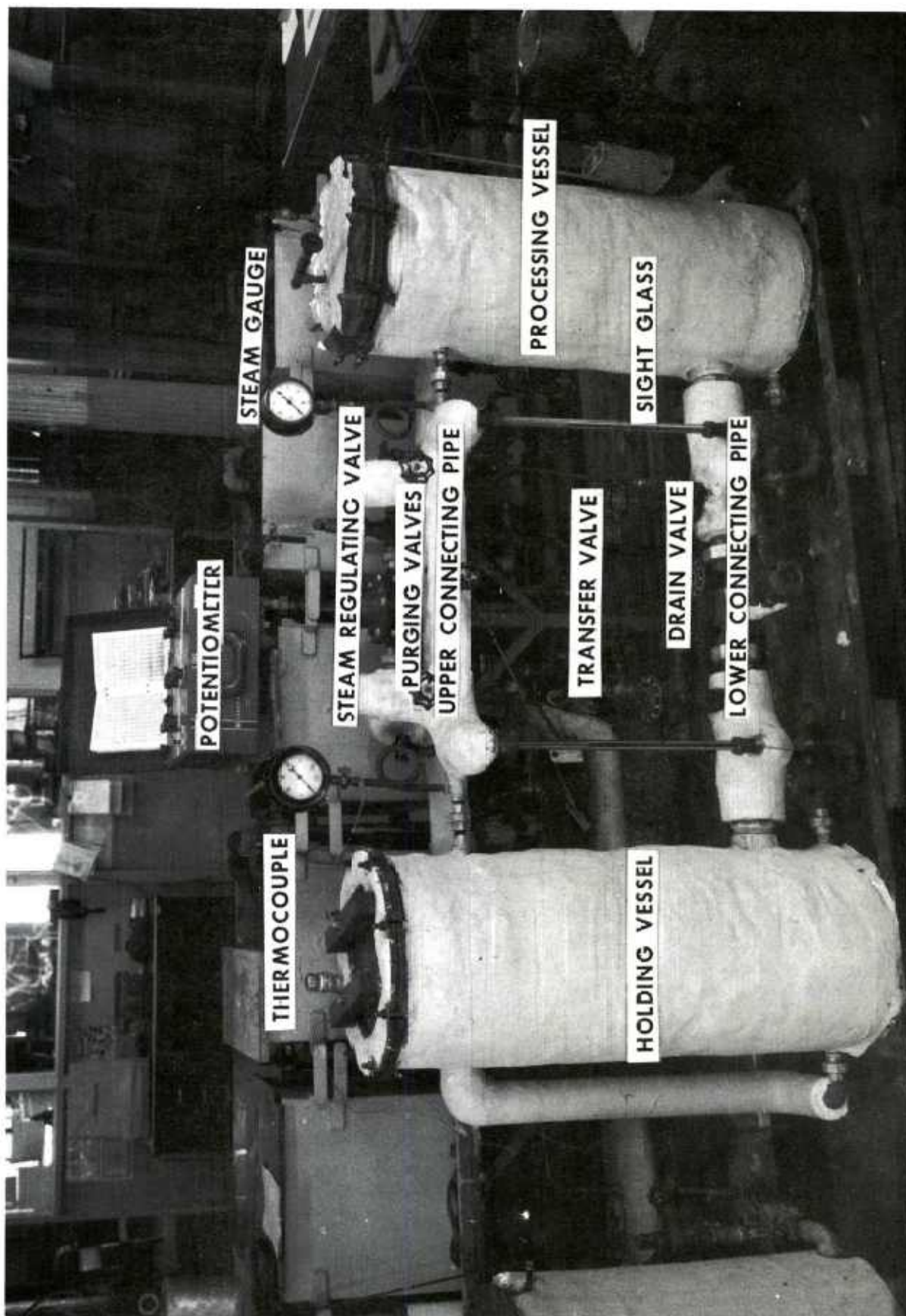


FIGURE 1

DUAL VESSELS FOR PHOSPHATING UNDER STEAM PRESSURE

- b. Either vessel can be used as a phosphating or holding tank.
- c. The bath can be maintained continuously under pressure.
- d. Sludge can be removed from the bath during the transfer of solution to the other vessel.
- e. The bath can be used continuously on a production basis.

The vessels are joined together by a two-pipe system. An upper 1/2 inch pipe connecting the vessels near the top is used to purge the system and equalize the steam pressure. The lower 1-1/2 inch pipe has two valves which can be operated manually to control the flow of the solution between the vessels under pressure. This pipe joins the stainless steel inner processing chambers. A steam gauge and a sight glass are attached to the system for monitoring pressure and liquid level. The liquid level can thus be observed during the transfer of the solution. A steam regulating valve is used to control the flow of steam to the inlet line connected at the base of the vessels. Steam traps at the base of the vessels control the flow of condensate.

The stainless steel liner in each vessel has a capacity of 8.0 gallons (30 liters). Each of the containers and the phosphating solution are heated with steam. The processing procedure was as follows: Steel panels (SAE 1020, 2 x 3 x 1/16 inches) were blasted with #80 steel grit. They were then suspended on a rack and placed in the phosphating bath at 170°F to 180°F. A constantan-copper thermocouple was inserted into the bath and connected externally to a potentiometer. The lid was bolted in place, and the steam turned on. Air was purged from the vessel, and the purging valve was closed. Steam pressure from 1 to 1-1/2 pounds per square inch gauge (psig) was used to raise the temperature of the bath to 212° - 213°F. Steam pressure was then maintained at 1 psig so that the temperature of the bath remains constant. The time of processing was continued for an additional 15 minutes. The bath was then transferred to the other vessel under a slight differential in steam pressure. Pressure on the phosphating vessel was released and the lid was removed. The coated panels were placed in the rinse tank. Recycling of the bath was accomplished by increasing the steam pressure of the holding vessel thereby forcing the bath to return to the processing vessel.

For chemical analyses, free acid and total acid determinations were done on 10-milliliter samples of the bath. These determinations were done by titration with sodium hydroxide (NaOH) to a phenolphthalein endpoint for free acid and a combination methylorange-xylene cyanole for the total acid. Values are listed in points (milliliters) of 0.1N NaOH to reach the endpoint for a 10 milliliter sample.

To control the free acid content of the bath, manganese carbonate (MnCO_3) was added. This addition was usually necessary after each run since the free acid value would rise after processing. The addition of manganese dihydrogen phosphate $[\text{Mn}(\text{H}_2\text{PO}_4)_2]$, served as a replenisher for the loss of manganese and phosphate as coating and sludge during processing.

Four sets of evaluations were conducted on the dual vessel line using different chemical additives. These included: manganese citrate, manganese gluconate, manganese tartrate additions and no manganese-organic compound addition.

The coating weight of panels, an important value for assessing coating quality, was determined by weighing the panel with the coating and subtracting the weight of the panel with the coating removed by stripping. Stripping was accomplished by immersion of the panel for 15 minutes in chromic acid solution heated to 180°F .

Salt spray testing was used to determine corrosion resistance. The testing was done according to ASTM (B117). Visual examination of the panels labeled the coatings a corrosion failure if three or more rust spots appeared. For comparative purposes, an arbitrary standard of 100 hours salt spray resistance was selected to denote a successful protective coating. Salt spray testing was divided into two groups: unheated panels and panels heated to 450°F for one hour. Heating was done in an oven at atmospheric conditions.

RESULTS AND DISCUSSION

Presented are the results of consecutive phosphating operations used to test the production-type dual-chamber pressure phosphating apparatus. Tables 1 through 8 display the phosphating results and analyses for each of the four types of baths. Specifically, Tables 1, 3, 5 and 7 show the chemical analyses (free acid and total acid bath content before processing), and chemical additives (additions of manganese dihydrogen phosphate, manganese carbonate, and manganese-organic compound to the conventional bath). Tables 2, 4, 6 and 8 show the physical characteristics of the coating. They display the weight of the phosphate coating in milligrams per square foot and the salt spray corrosion resistance in hours for both heated and unheated panels.

The first series of tests were conducted with manganese citrate additions to the bath. These tests were done in two separate cycles of three and four consecutive phosphating runs (Table 1). Salt spray results presented in Table 2 show only 14% of the heated and 29% of the unheated sets of panels passing the 100-hour minimum salt spray corrosion test.

TABLE 1

CHEMICAL CONTROL OF MANGANESE PHOSPHATE BATH ENRICHED WITH MANGANESE CITRATE

Cycle and Processing Run	Analyses (points NaOH)		Additives (grams)		
	Free Acid	Total Acid	$\text{Mn}(\text{H}_2\text{PO}_4)_2$	MnCO_3	Mn-citrate
<u>Cycle A</u>					
1	3.2	37.6	60	90	2
2	4.3	33.9	120	120	3
3	4.3	35.4	-	-	4
<u>Cycle B</u>					
1	3.1	39.0	-	200	-
2	4.0	31.3	200	200	1
3	3.2	30.0	300	200	1
4	4.2	30.3	-	-	2
AVERAGE	3.8	33.9	97	116	2

TABLE 2

PHYSICAL PROPERTIES OF MANGANESE PHOSPHATE COATINGS ENRICHED WITH MANGANESE CITRATE

Cycle and Processing Run	Weight of Coating (milligrams per square foot)	Salt Spray Tests (hours)	
		Heated 450°F	Unheated
<u>Cycle A</u>			
1	5968	2	4
2	6945	2	4
3	2647	2	4
<u>Cycle B</u>			
1	4504	4	4
2	5865	4	720
3	6948	720	720
4	4183	2	2
AVERAGE		5294	

TABLE 3

CHEMICAL CONTROL OF MANGANESE PHOSPHATE BATH ENRICHED WITH MANGANESE GLUCONATE

Cycle and Processing Run	Analyses (points)		Additives (Grams)		
	Free Acid	Total Acid	Mn(H ₂ PO ₄) ₂	MnCO ₃	Mn-gluconate
<u>Cycle A</u>					
1	3.4	31.0	-	-	10
2	3.8	30.1	100	100	10
3	4.7	28.5	400	350	10
4	3.5	27.1	200	-	11
5	4.1	38.0	200	350	11
6	2.2	26.2	200	100	12
7	3.5	30.3	200	300	12
8	3.3	27.1	-	-	13
<u>Cycle B</u>					
1	4.2	34.7	200	200	10
2	3.3	30.1	200	100	11
3	3.8	31.7	200	200	11
4	4.3	30.8	420	150	12
5	3.6	35.4	210	150	13
6	3.5	33.2	-	-	14
<u>Cycle C</u>					
1	2.9	25.3	-	-	10
2	3.3	26.3	120	120	11
3	2.8	29.4	150	150	12
4	4.4	29.5	180	180	12
5	3.4	28.8	210	210	12
6	3.0	27.9	210	210	13
7	2.2	24.6	210	210	14
8	2.2	26.0	-	-	15
AVERAGE	3.4	29.6	164	140	12

TABLE 4

PHYSICAL PROPERTIES OF MANGANESE PHOSPHATE COATINGS
ENRICHED WITH MANGANESE GLUCONATE

Cycle and Processing Run	Weight of Coating	Salt Spray Tests (hours)	
	(milligrams per square foot)	Heated (450°F)	Unheated
Cycle A			
1	5359		
2	4378	100	100
3	5876	5	3
4	1469	775	150
5	7886	1	3
6	9288	775	768
7	9347	775	720
8	6289	775	720
		672	744
Cycle B			
1	9047		
2	6953	744	432
3	8412	744	744
4	6509	744	744
5	10374	744	744
6	6676	744	744
		48	744
Cycle C			
1	7078		
2	1448	840	840
3	5189	1	1
4	1272	24	5
5	1659	1	1
6	7466	1	1
7	6430	600	100
8	6176	1	5
		1	5
AVERAGE	6117		

TABLE 5
CHEMICAL CONTROL OF MANGANESE PHOSPHATE BATH ENRICHED WITH MANGANESE TARTRATE

Processing Run	Analyses (points NaOH)		Additives (grams)		
	Free Acid	Total Acid	Mn(H ₂ PO ₄) ₂	MnCO ₃	Mn-tartrate
1	3.2	32.3	100	160	10
2	4.4	31.7	200	260	11
3	4.1	32.1	200	200	11
4	2.7	29.8	200	-	12
5	3.4	31.7	200	200	13
6	2.4	30.6	100	100	14
7	3.5	29.4	200	200	15
8	2.8	24.8	200	200	16
9	2.6	26.6	200	200	17
10	2.8	25.9	200	200	18
11	2.6	27.3	200	200	18
12	2.6	29.4	240	200	19
13	2.8	27.2	240	200	19
14	2.6	21.1	300	-	20
15	3.4	26.9	-	60	21
16	3.4	26.2	210	180	22
17	3.3	25.8	210	240	22
18	2.6	29.0	-	90	23
19	3.4	25.9	-	60	23
20	2.7	23.8	-	-	24
21	2.7	22.4	-	-	24
AVERAGE	3.0	27.6	152	140	18

TABLE 6
PHYSICAL PROPERTIES OF PHOSPHATE COATINGS ENRICHED
WITH MANGANESE TARTRATE

Processing Run	Weight of Coating (Milligrams per square foot)	Salt Spray Tests (hours)	
		Heated (450°F)	Unheated
1	8107	840	840
2	4109	30	24
3	6049	6	6
4	9497	840	336
5	7537	840	336
6	8640	336	336
7	7475	840	30
8	7417	840	840
9	9825	840	840
10	11030	840	840
11	8252	840	120
12	9309	840	840
13	7380	840	100
14	6582	840	840
15	5935	840	100
16	7268	840	120
17	7735	840	840
18	10360	840	840
19	6970	100	408
20	9314	840	840
21	5182	168	72
AVERAGE	7808		

TABLE 7

CHEMICAL CONTROL OF MANGANESE PHOSPHATE BATH ENRICHED WITH MANGANESE DIHYDROGEN PHOSPHATE

Processing Run	Analyses (points NaOH)		Additives (grams)		
	Free Acid	Total Acid	Mn(H ₂ PO ₄) ₂	MnCO ₃	Mn-organic compound
1	1.0	23.5	159	60	none
2	1.4	23.5	155	130	none
3	1.8	22.2	120	90	none
4	1.3	22.0	145	90	none
5	1.0	28.7	465	120	none
6	1.6	26.4	150	120	none
7	1.4	23.4	90	60	none
8	1.4	24.3	150	60	none
9	0.5	23.4	200	150	none
10	1.0	24.5	300	180	none
AVERAGE	1.2	24.2	193	106	

TABLE 8

PHYSICAL PROPERTIES OF MANGANESE PHOSPHATE COATINGS ENRICHED WITH MANGANESE DIHYDROGEN PHOSPHATE

Processing Run	Weight of Coating (milligrams per square foot)	Salt Spray Tests (hours)	
		Heated 450°F	Unheated
1	7287	408	336
2	8159	264	100
3	5561	48	5
4	8436	264	48
5	6395	240	100
6	6972	840	408
7	7034	840	8
8	8436	840	100
9	6983	840	240
10	8020	840	264
AVERAGE	7328		

The second series of tests used manganese gluconate additions. These tests were conducted in three separate cycles of 8, 6 and 8 processing runs (Table 3). Salt spray results given in Table 4 show 59% of the heated and 64% of the unheated sets of panels passing a 100-hour minimum salt spray test.

The third series of tests were conducted with manganese tartrate additions to the bath. In this test 21 consecutive runs were performed on the same bath (Table 5). Salt spray results in Table 6 show 90% of the heated and 81% of the unheated sets of panels passing the minimum 100-hour salt spray test.

Finally, testing was conducted without any manganese-organic compound addition to the bath. Only manganese dihydrogen phosphate for bath replenishment and manganese carbonate for free acid control were used (Table 7). For this bath, ten consecutive runs were conducted. Salt spray results of Table 8 show 70% of the heated and 90% of the unheated sets of panels passing a 100-hour minimum salt spray test.

The manganese citrate phosphating results were the poorest. Low salt spray corrosion resistance can probably be attributed to high initial free acid and insufficient additions of manganese compounds. The average initial free acid content of this bath was 3.8 points. This is higher than the 3.4, 3.0 and 1.2 point averages of the other baths. A high free acid bath content will produce a thinner phosphate coating, and consequently this coating will provide less protection against corrosion. Also, the amount of manganese citrate added to the bath is small when compared to the amount of manganese organic compound added to the manganese gluconate and tartrate baths. Initially for this project, small quantities of the manganese-organic compound were added to the phosphating bath. This was the case for the Mn-citrate bath. Later, for the Mn-gluconate and Mn-tartrate baths, larger amounts were added to improve the coatings and obtain better corrosion resistance. The amount of manganese dihydrogen phosphate added is also lower than the amount added to the other baths. This lower bath concentration of $\text{Mn}(\text{H}_2\text{PO}_4)_2$ results in thin coatings of inferior corrosion resistance. This combination of high free acid and low manganese compound content yields coatings for this bath that are unable to meet a 100-hour minimum salt spray test.

Results from the manganese gluconate bath varied. Cycles A and B produced good coatings, while Cycle C yielded poor coatings. A plausible explanation for the poor corrosion resistance of the coatings produced from Cycle C is the low initial total acid content. This low value of total acid means insufficient manganese dihydrogen phosphate in the bath. Thus thin coatings with poor corrosion resistance are produced. The minimum value of initial total acid should be at least 30 points for reproducibly good coatings.

The results from the tartrate bath are the best for illustrating the success of pressure phosphating using consecutive runs in the dual vessel system. The bath produced the greatest percentage of sets of panels passing a minimum 100 hours in the salt spray. Additionally, 76% of the heated and 43% of the unheated sets of panels exceed 800 hours of salt spray testing. A total of 21 consecutive processing periods demonstrated the success of production-type pressure phosphating.

The bath without any manganese-organic compound addition was also successful. Larger additions of manganese dihydrogen phosphate were substituted for the Mn-organic compound additions. The consecutive number of runs could have been extended; however, ten consecutive runs were considered sufficient to illustrate the success of the bath and procedure.

These series of tests conducted on the dual vessel phosphating system show that successful production-type pressure phosphating is possible. Limitations for the process exist with the large amount of precipitate deposited after processing. A sludge recovery unit for converting the precipitate to the useful form of manganese dihydrogen phosphate would most likely need to be installed for the system to be production feasible. As shown by the results, careful control of the bath is also needed to produce good quality coatings. Initial free and total acid must be carefully controlled. The bath must also be maintained with chemical additions after every processing period to replenish the depleted bath. With proper maintenance of the bath, the reproducibility of pressure phosphate coatings having superior heat and corrosion resistance should be easily obtained.

CONCLUSIONS

1. Manganese phosphate coatings of high quality can be applied consecutively under carefully controlled conditions on ferrous metal items processed in an enriched bath under low steam pressure.
2. A phosphating bath operated continuously under constant pressure requires frequent replenishment and acid control to function properly. To operate the bath efficiently, automated methods are needed to maintain the bath with a total acid value exceeding 30 points and a free acid value less than 3.5 points.
3. The success of pressure phosphating in the dual vessel system is shown by the salt spray corrosion results of the manganese tartrate bath. This bath had 21 consecutive processing periods and yielded 90% of the heated and 81% of the unheated sets of panels passing the minimum 100-hour salt spray test. Additionally, 76% of the heated and 43% of the unheated sets of panels surpassed 800 hours of salt spray testing.

RECOMMENDATIONS

It is recommended that:

1. The method of phosphating ferrous metal items in a bath enriched with manganese compound additives (particularly, manganese tartrate and excess manganese dihydrogen phosphate) and processed under steam pressure be utilized for the application of heavy manganese phosphate coatings.
2. A sludge recycling system be designed for the pressure phosphating system so that the precipitate can be converted to the useful form of $\text{Mn}(\text{H}_2\text{PO}_4)_2$.
3. The small 30 liter production-type dual vessel system should be scaled-up to manufacturing level capacity.

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A DUAL VESSEL SYSTEM OF PHOSPHATING FERROUS ALLOYS UNDER STEAM PRESSURE, by Henry Crain and Linden H. Wagner

Report EN-79-03, Aug 79, 21 p. incl. illus. tables, (AMS Code 3297.16.6785) Unclassified report.

A dual vessel system of phosphating ferrous alloys under steam pressure was investigated for the application of high temperature corrosion resistant coatings on a continuous production-type basis. In this system, the phosphating bath can be transferred from one vessel to another using differential steam pressure. Either vessel can be used as a phosphating or holding tank and the bath can be maintained continuously under pressure. Sludge can be removed from the bath during (Cont.) over

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